

# Possible Exhaust Gas Aftertreatment Concepts for Passenger Car Diesel Engines with Sulphur-free Fuel

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## ABSTRACT

In order to fulfill future emissions standards, there is a need for new exhaust-gas aftertreatment concepts, with NO<sub>x</sub>-emissions reduction in passenger car diesel engines being of particular importance. The NO<sub>x</sub> storage catalyst is one of the technologies currently under discussion with high NO<sub>x</sub> conversion potential, and which is under development at DaimlerChrysler for EURO IV standards. With this system, the nitrogen oxides contained in the diesel exhaust gas are stored under lean exhaust-gas conditions and are reduced in the catalyst through an enriched air-fuel ratio of the exhaust-gas and favorable thermal conditions. Hydrocarbons, carbon monoxide and hydrogen are used as reducing agents.

DaimlerChrysler has analyzed the effect of sulphur contained in the fuel on the operation of various catalysts during laboratory and engine testing. The sulphur dioxide in the exhaust gas generates sulfates, which remain on the catalyst when nitrate compounds are regenerated briefly. For this reason, NO<sub>x</sub> conversion efficiency deteriorates rapidly. Using the diesel fuel currently available in Europe with a sulphur content of 370 ppm (limit: 500 ppm), the efficiency of the catalyst is significantly reduced within a few operating hours.

However, using diesel fuel with a sulphur content of 10 ppm in a C220 CDI fitted with a NO<sub>x</sub> storage catalyst, a potential for NO<sub>x</sub> reduction in the New European Driving Cycle (NEDC) of over 50% could be achieved in repeated testing.

## INTRODUCTION

Sulfur is a natural component of mineral oil and is essentially available in the form of mercaptanes, chain and cyclic sulfides as well as heterocyclic aromatic hydrocarbons. The last form, alkylated benzothiophenes, is the main sulfur carrier in the case of diesel fuel. In general, the proportion of sulfur-containing hydrocarbons increases as the boiling range of the mineral oil fraction increases. This is the reason why the sulfur content is usually higher in diesel fuel than in gasoline. The respective sulfur content of mineral oil depends greatly

on the location of the oil fields. For example the mineral oil from the oil fields in the North Sea has approx. 0.2 mass percent sulfur and crude oil from the Arabian oil fields has approx. 1.0 mass percent /1/.

The influence of sulfur contained in diesel fuel on emissions and component loads has already been examined and evaluated extensively due to its great importance /2,3/. **Figure 1** gives an overview of this. The greater part of the fuel sulfur is oxidized during combustion to sulfur dioxide SO<sub>2</sub>. The by-products this creates in the atmosphere, SO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub>, are environmentally relevant because of their contribution to acid rain.

<b>Emissions</b>	<ul style="list-style-type: none"> <li>- Formation of sulphur dioxide SO<sub>2</sub></li> <li>- SO<sub>3</sub> and H<sub>2</sub>SO<sub>4</sub> are involved in causing "acid rain"</li> <li>- Formation of sulphates and adsorption on soot-particulates</li> </ul>
<b>Corrosion and Wear</b>	<ul style="list-style-type: none"> <li>- Increasing wear of engine parts through the corrosion by internal formed acid</li> <li>- Corrosion in the exhaust system through sulphuric acid and condensate accumulation</li> </ul>
<b>Exhaust Aftertreatment</b>	<ul style="list-style-type: none"> <li>- Formation of sulphates in the oxidation catalyst and therefore increasing of particulate matter</li> </ul>

What's the effect of the sulphur in the fuel for the future

DENOX-Technologies?

**Figure 1:** Effect of sulphur on diesel fuel

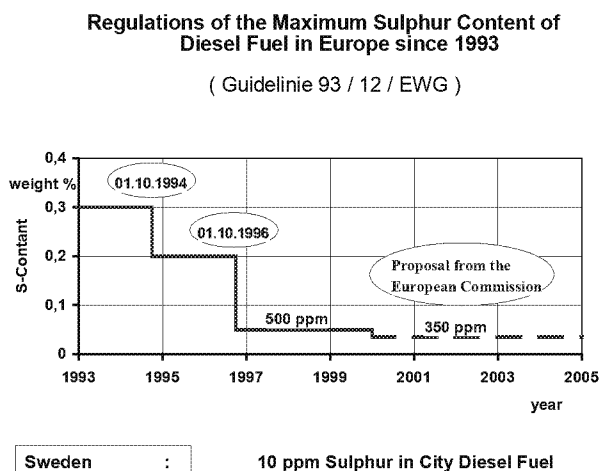
The remaining part of the sulfur contained in fuel is emitted in the exhaust gas in the form of sulfates, which partially accumulate on the soot particles created during combustion and therefore have decisive influence on the total particle emission of the diesel engine. Previous investigations showed a doubling of the particle emissions in the driving cycle prescribed for the certification of passenger cars when diesel fuel at 450 ppm was used compared to a driving cycle with

sulfur-free diesel fuel /2/. It is also known, that sulfur content and particle emission are strongly correlated /3/.

Under cold-start conditions, sulfur-containing acids are formed which cause an increase in wear due to corrosive attacks on the cylinder liners. When the dew-point temperature is not reached, the sulfuric acid created in the exhaust gas tract leads to increased corrosion of individual components in the exhaust gas system. High demands are therefore placed on the materials used, especially in locations where condensed water collects, such as in the exhaust gas recirculation system.

The influence of sulfur contained in fuel on the particle emission of a passenger car diesel engine using an oxidation catalyst is examined and described in /2/. There is then an increase in total particle emissions caused by additional sulfate formation in the oxidizing catalytic converter. This effect occurs in particular in the case of operating points with high exhaust gas temperature.

Because of the environmental relevance of SO<sub>x</sub> emissions caused by the fuel sulfur, the maximal permissible sulfur content in diesel fuel has been reduced in Europe gradually since 1960. At present, guideline 93/12/EWG of 01.10.94 is valid, with a maximum sulfur content of 0.2 mass percent, reduced to a maximum of 0.05 mass percent (which corresponds to 500 ppm) since 01.10.96 as shown in **Figure 2**.



**Figure 2:** Sulphur content of diesel fuel

In some markets, diesel fuel with a maximum sulfur content of 10 ppm is already offered (e.g. Sweden). This shows clearly that the current boundary value of 500 ppm is far in excess of the current technically feasible value.

In addition to the reduction of vehicle emission limits in 2000 and 2005, an improvement in fuel quality is above all necessary to improve air quality. The sulphur content limits proposed by government and European

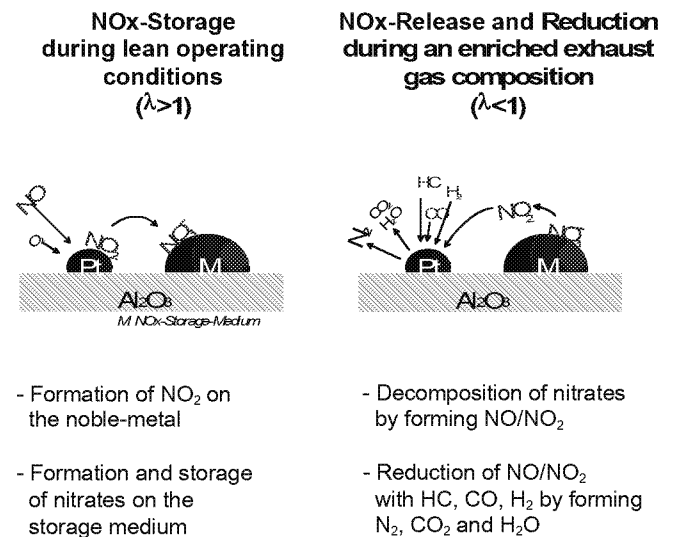
authorities, ranging from 50 ppm to 350 ppm, must be rejected as insufficient. The target must be a maximum of 10 ppm sulphur content in fuel.

The subject of the results described in the following sections, is the influence of sulfur contained in fuel on NO<sub>x</sub> storage catalytic converter technology under diesel engine conditions.

## NO<sub>x</sub> STORAGE CATALYTIC CONVERTER TECHNOLOGY

### OPERATING PRINCIPLES AND POTENTIAL ESTIMATE

In order to fulfil future emission limits, new methods are necessary for exhaust gas aftertreatment, especially with regard to NO<sub>x</sub> emissions from passenger car diesel engines. NO<sub>x</sub> storage catalytic converter technology is one of the procedures currently under discussion which demonstrates a high NO<sub>x</sub> reduction potential. The NO<sub>x</sub> storage catalytic converters which were at first developed for the lean-burn gasoline engine, contain a basic NO<sub>x</sub> storage component in addition to noble metals for the three-way function. The operating principle of the NO<sub>x</sub> storage catalytic converter is illustrated in **Figure 3**. In an oxidizing atmosphere with lean exhaust gas composition, the nitrogen oxides emitted by the engine are oxidized on the noble metal substrate and then stored in a further oxidation step through the formation of nitrates by reaction with the basic components.



**Figure 3:** Operating principle of the NO<sub>x</sub>-storage catalyst technology

Under the reducing reaction conditions in the case of rich exhaust gas composition, the nitrates can be decomposed again and the nitrogen oxides which are released are reduced to N<sub>2</sub> at the noble metal substrate. In this case, the reducing agent is hydrocarbons, carbon monoxide or hydrogen. This catalytic converter principle,

which was first published by Toyota /6,7/, is a discontinuous procedure and requires controlled transitions between oxidizing and reducing exhaust gas atmospheres.

The NOx storage catalytic converter is effective only in a limited temperature window and therefore experiences a problem zone both at very low and at high exhaust gas temperatures. The lower temperature limit is determined by oxidation of NO to NO<sub>2</sub> at the noble metal contact. On the other hand, the upper temperature limit is determined by the thermal stability of the nitrates. Accordingly, the overall NOx conversion efficiency depends greatly on the operating temperature of the catalyst. It is also important that the stoichiometry of the reactions is taken into consideration and that reduction agent supply is high enough for the stored nitrates and the oxygen stored in the "washcoat".

The high NOx reduction potential of the NOx storage catalytic converter has already been demonstrated with the lean operated gasoline engine /8,9,10,11/.

For diesel applications, different types of NOx storage catalytic converters with different compositions were examined in laboratory tests. In this case, cycles of 90s with lean and 4s with rich synthetic exhaust gas at a space velocity of 30000 h<sup>-1</sup> and at constant temperature were performed. The composition of the synthetic exhaust gas corresponded to the diesel engine exhaust gas without sulphur dioxide.

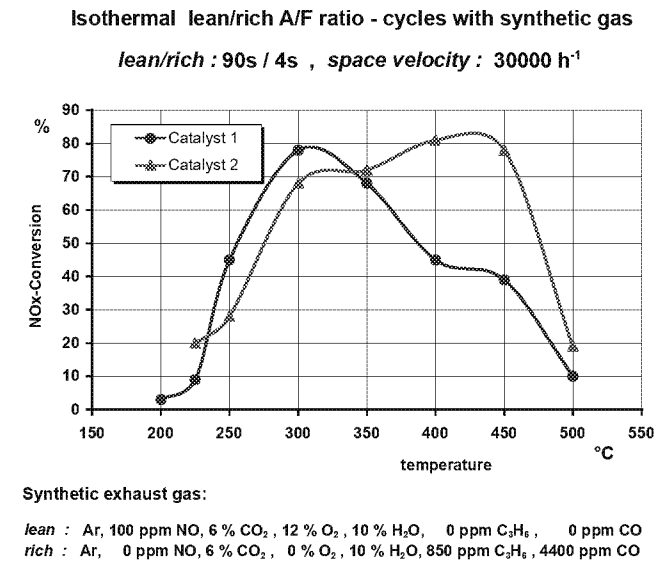


Figure 4: Potential of NOx-storage catalysts

Figure 4 shows the results of these laboratory tests for two different catalysts. Of particular note is the broad operation range of 250°C to 500°C for both catalyst models. The conversion maxima occur at different temperatures according to the different compositions.

The high conversion rates of up to 80 % show a far higher potential for this technology compared to continuous-operation DENOX catalytic converters.

OPERATIONAL FEASIBILITY OF NOx STORAGE TECHNOLOGY IN DI DIESEL ENGINES

The implementation of the NOx storage technology in the diesel engine is dependent on a rich exhaust gas composition for the reduction of the nitrogen oxides under different operating conditions can be achieved in reproducible form.

Due to the heterogeneous diesel fuel mixture in the entire operating range passenger car diesel engines work with air surplus. Accordingly, a lean exhaust gas composition is constantly available. In this case, once a NOx storage catalytic converter in the exhaust system has reached the light-off temperature it adsorbs nitrogen oxides from the exhaust gas until its storage capacity is exhausted.

For desorption of the stored nitrates and simultaneous deoxidization of nitrogen oxides it is necessary to ensure a short-term rich exhaust gas composition in the diesel engine exhaust gas. Figure 5 includes a summary of the possibilities currently under discussion for producing a rich exhaust gas composition before the storage catalytic converter.

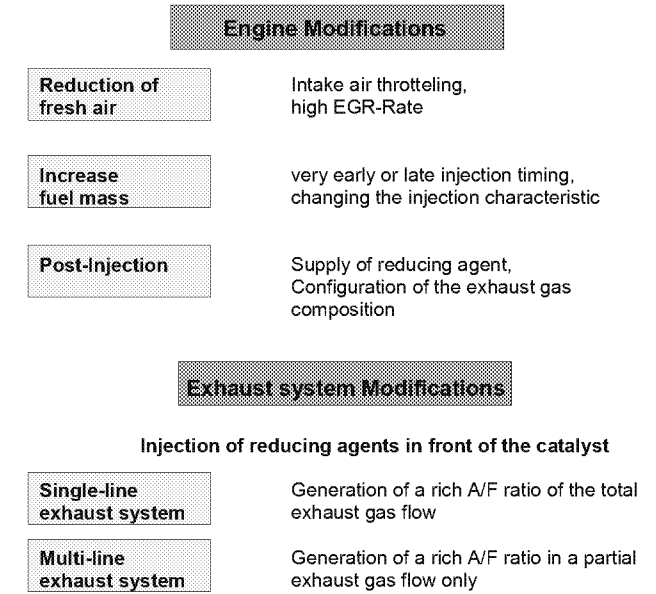


Figure 5: Realization of an enriched exhaust gas composition

A distinction can be made between engine internal and engine external measures. Internal measures include the extreme pre-shift of combustion /12/. Predominantly pre-mixed combustion is generated by additional pre-injection during the compression stroke and simultaneous measures for the reduction of the intake air flow. This leads to a reduction in the thermal efficiency of the engine and the combustion air-/ fuel ratio is lowered.

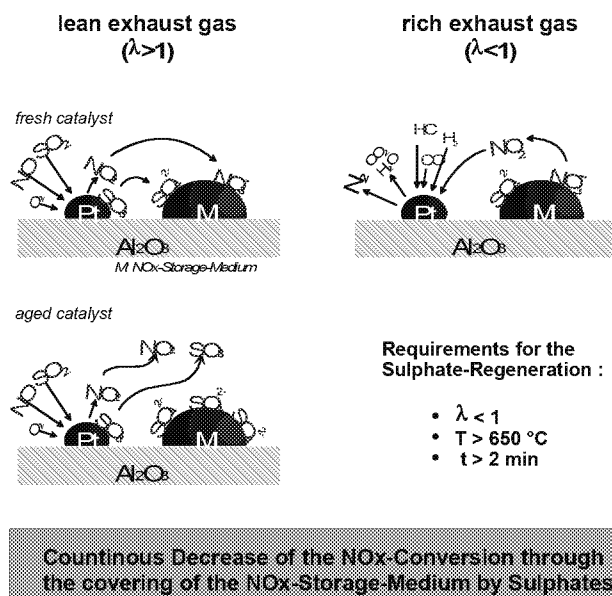
Similar effects on efficiency and combustion air-/ fuel ratio can be obtained by shifting the combustion far after top dead center by retarding the injection timing and simultaneously reducing the intake air mass. A further internal measure to lower the total air-/ fuel ratio is the use of post-injection in the combustion chamber /13/ which is not participant in the combustion process, by means of electronically controlled injection systems. Reducing agent content as well as the exhaust gas temperature of the exhaust gas composition can be controlled by altering the injection-timing and the rate of post-injection into the combustion chamber.

It is also possible to make use of external measures, such as to inject reducing agent into the exhaust gas system and by so doing to generate a rich exhaust gas composition upstream of the catalyst. A two-flute exhaust system with a NOx storage catalytic converter in at least one flute is also possible /14/. For the desorption of the nitrates and reduction of nitrogen oxides, this flute is isolated to a large extent from the total exhaust gas flow, and a reducing exhaust atmosphere is achieved by injection of reducing agents upstream of the catalyst.

## INFLUENCE OF SULFUR ON NOX STORAGE TECHNOLOGY

### CHEMICAL FUNDAMENTALS

The SO<sub>2</sub> contained in the exhaust gas is also oxidized to SO<sub>3</sub> at the noble metal contact in competition with the nitrogen oxides. The formation and accumulation of sulfates in the storage catalytic converter occurs in a similar way to how the nitrates are formed and accumulated. This formation mechanism is represented schematically in **Figure 6**.



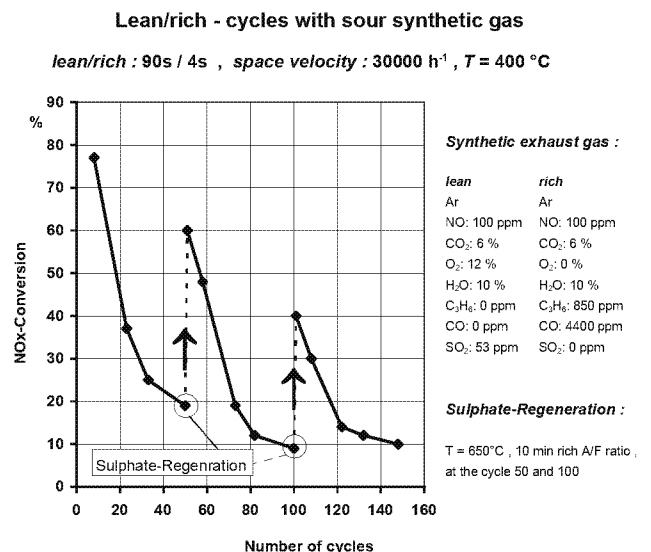
**Figure 6:** Effect of sulphur in the fuel on the catalyst surface

Compared to the nitrates, the stored sulfates are considerably more stable thermodynamically. This means that the sulfates do not decompose during the short-term nitrate regeneration in the catalytic converter and remain in the catalytic converter. The NOx storage capacity is reduced by continued poisoning of the storage components and the catalytic converter is increasingly deactivated.

Unlike the nitrate regeneration, the reaction conditions for a sulfate regeneration are much more extreme. In an oxidizing exhaust gas atmosphere, thermal disintegration is not possible because of the thermodynamic stability of sulfates under real driving conditions. Even under rich exhaust gas conditions, exhaust gas temperatures in excess of 650°C are necessary. Additionally these reaction conditions must be fulfilled over a much longer period than for nitrate regeneration.

### LABORATORY RESEARCH WITH SYNTHETIC EXHAUST GAS

The influence of SO<sub>2</sub> on a typical NOx storage catalytic converter was examined in the lab in "lean/rich"-cycles of 90s/4s at a constant temperature of 400°C and a space velocity of 30000 h<sup>-1</sup>. The lean gas composition corresponds to a simplified, however diesel-typical exhaust gas with NO, CO<sub>2</sub>, H<sub>2</sub>O and O<sub>2</sub>. In addition the synthetic exhaust gas contains 53 ppm of SO<sub>2</sub>, which corresponds to a sulfur content in the fuel of approx. 750 ppm at a combustion air/fuel ratio of  $\lambda = 1$ . The rich synthetic exhaust gas was created by switching off the O<sub>2</sub> with correspondingly high proportions of CO and HC.



**Figure 7:** Laboratory test of the effect of sulphur on the NOx-conversion of a NOx-storage catalyst

In **Figure 7**, it can be seen that after just 50 cycles (approx. 75 min) the attainable NOx reduction of 80% initially drops under the chosen test conditions to approx. 20%. After 50 cycles, a sulfate regeneration was carried

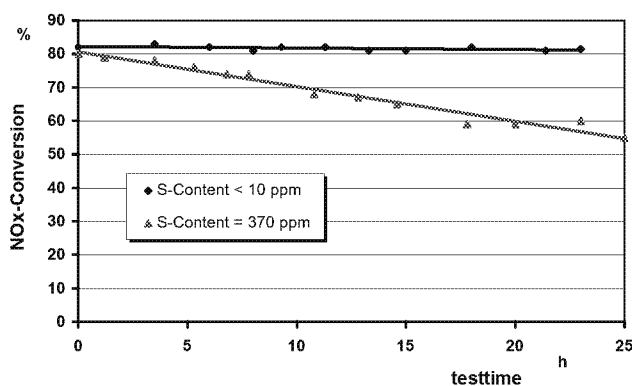
out by feeding the catalytic converter for 10 minutes with a rich gas composition at a temperature of 650°C. As a result, NO<sub>x</sub> activity was increased again to 60%. However, the initial value of 80% could not be achieved. In the following 50 cycles, NO<sub>x</sub> conversion declined to approx. 10%. After 100 cycles, a further sulfate regeneration was carried out, meaning the NO<sub>x</sub> reduction rate in the following cycle could once again be increased to 40%. Subsequently, a rapid decrease of NO<sub>x</sub> conversion is again observed.

The tests show clearly that the sulfur has an extremely harmful influence. Deactivation due to sulfate formation is a poisoning process which progresses very rapidly and leads to an unacceptable decrease in NO<sub>x</sub> conversion efficiency. Despite long regeneration times and extreme reaction conditions, the tested catalytic converter could not be reset again to the initial level. This means that even after 10 min sulfate regeneration was not complete.

#### INVESTIGATIONS WITH REAL DIESEL EXHAUST GAS IN THE PARTIAL CURRENT

The investigation below compares standard diesel fuel with a sulfur content of 370 ppm with Sweden city diesel at 10 ppm sulfur in the fuel. For these tests an experimental catalytic converter system was used on the engine test stand. The advantage of this system is that the catalytic converter samples can be examined under precisely defined conditions, as in the laboratory, but real exhaust gas is used.

**Lean/rich cycles in a partial exhaust flow (Engine: OM602DE29LA)**  
lean/rich : 3min / 1min , SV : 30000 h<sup>-1</sup> , T = 350 °C (const.)



**Adsorption** : Catalyst supplied with real Diesel exhaust gas

**Regeneration** : Catalyst supplied with synthetic gas out of N<sub>2</sub> with 0,9 % CO

**Figure 8:** Aging of the NO<sub>x</sub>-storage-catalyst in a partial exhaust flow by sulphur

During the storage phase, the catalyst was fed with the exhaust gas of a DI diesel engine 3 minutes each time at a constant temperature of 350°C and a space velocity of 30000 h<sup>-1</sup>. For the subsequent regeneration phase, lasting 1 minute each time, a synthetic, reducing exhaust

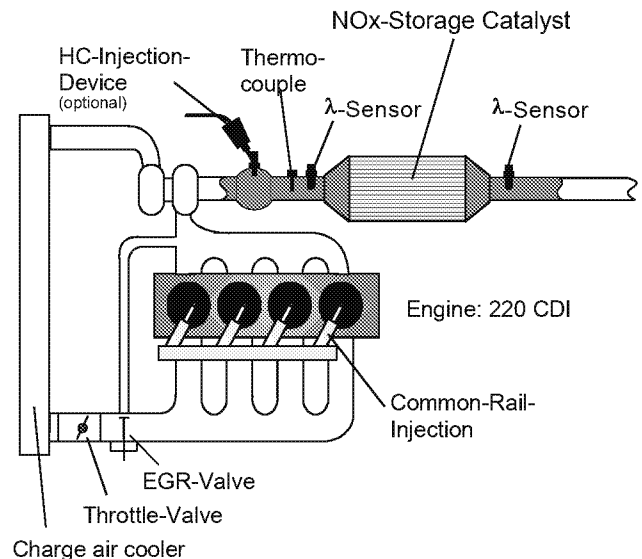
gas similar to that used in the laboratory was used. The temperature was held constant at 350°C during this by heating the catalyst electrically.

With a sulfur content of 370 ppm, the initial NO<sub>x</sub> conversion rate of 80% sank after 25 hours of operation to approx. 55% (**Figure 8**). With the Sweden city diesel, no drop in NO<sub>x</sub> conversion was noticeable after this short period.

These investigations display the poisoning influence of sulfur on NO<sub>x</sub> storage catalytic converter technology even using diesel fuel currently available in Europe with an average sulfur content of 370 ppm.

#### RESULTS ON THE ENGINE TEST STAND

For the further investigation of the potential of NO<sub>x</sub> storage technology, a NO<sub>x</sub> storage catalytic converter positioned close to the engine was installed in the exhaust system of an experimental engine on an engine test stand, as shown in **Figure 9**.

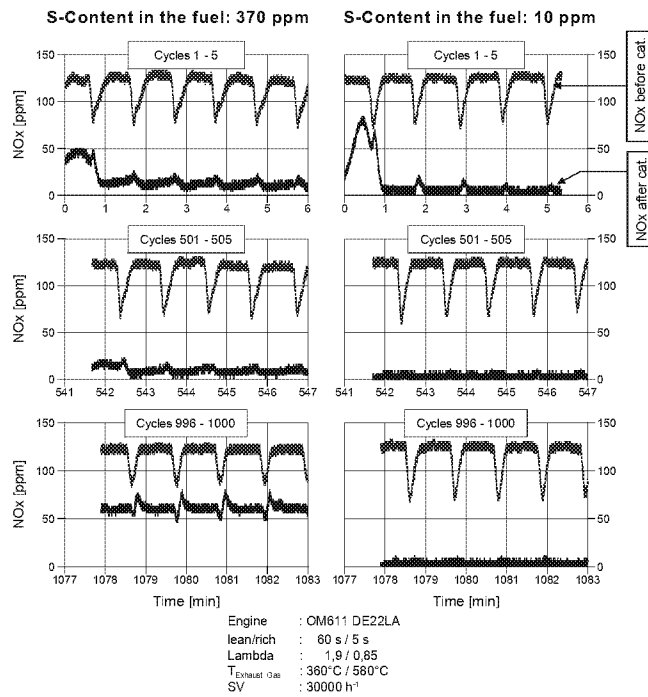


**Figure 9:** NO<sub>x</sub>-storage-system for diesel: Setup for a 4-cyl. DI diesel

In this case a 2.2 liter DI diesel engine with common rail injection system (220 CDI) was used. The NO<sub>x</sub> storage catalytic converter which was used for the tests has a volume of 2.1 liters. The reduction of the total air-/ fuel ratio for the regeneration phase was controlled by influencing the combustion process and decrease the intake air-mass via the electronic engine control unit. An optional unit for supplying reducing agents can help in setting the exhaust gas composition to the desired value. The temperature of the exhaust gas at the catalyst entrance which serves as a guide variable for the regeneration phase, was measured with a thermocouple arranged between turbine and catalyst. The measurement of the total air-/ fuel ratio was made before

and after the catalyst using a wide-band lambda sensor each time. The investigations were carried out at an engine speed of  $n = 1850$  rpm and a brake mean effective pressure of 2.3 bar. The catalyst was supplied alternately for 60s with a lean exhaust gas composition of  $\lambda = 1.9$  ( storage phase ) and for 5s with a rich exhaust gas mixture of  $\lambda = 0.85$  ( regeneration phase ).

For the investigations a standard diesel fuel with 370 ppm sulfur content and Sweden city diesel with 10 ppm sulfur content was used.



**Figure 10:** Sulphur-aging of a NOx-storage catalyst by lean/rich cycles in the real diesel exhaust gas engine

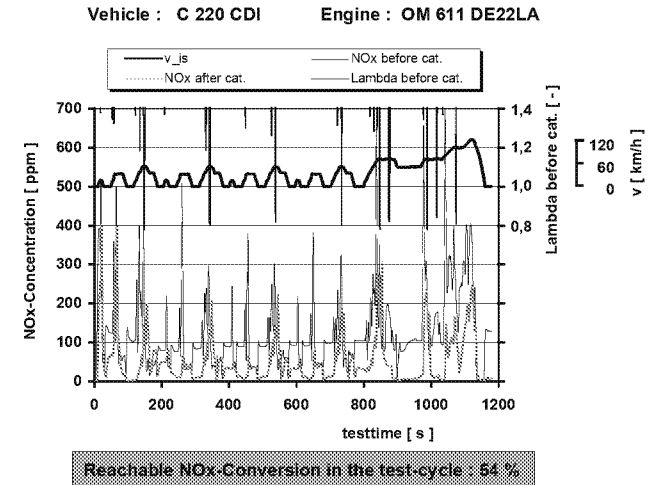
The result of the investigations on NOx conversion rates is represented schematically in **Figure 10** for the cycles 1 to 5, the cycles 501 to 505 and the cycles 995 to 1000. The regeneration stages are to be recognized by the respective drop of the NOx trace before catalytic converter entry. During this investigation, the influence of the sulfur content on the attainable NOx reduction can be clearly seen. After only 18 hours (1000 cycles) using diesel fuel with 370 ppm sulfur content, the NOx reduction sank to approx. 50% from an initial 80%. During the investigations with Sweden city diesel, no drop in the NOx reduction by the catalytic converter was registered in this time. The influence of the small sulfur part of 10 ppm in diesel fuel on the operating life of the storage catalytic converter will be examined in an endurance test.

## VEHICLE TEST RESULTS

Finally, the potential of the NOx storage catalytic converter was evaluated under transient conditions in a C-class vehicle, equipped in accordance with the

experimental setup described above. The tests on the roller test bench ran under the test conditions for EURO III certification and were carried out with Sweden city diesel (10 ppm sulfur content).

### NOx-Concentration before and after the NOx-Storage Catalyst in the NEDC with Regeneration cycles



**Figure 11:** Potential of the NOx-storage-technology in the New European Driving Cycle (NEDC)

In **Figure 11**, the NOx traces over the test time are depicted both in front of and after the storage catalytic converter in the NEDC cold test. Furthermore, the driving speed characteristics and the lambda signal in front of the catalytic converter, which is cut off at the top, can be seen. By lambda values  $< 1$  the regeneration stages are to be recognized. After every regeneration stage, the storage catalytic converter shows very good adsorptivity, which demonstrates almost complete regeneration. Several consecutive tests of this kind achieved NOx reductions of 50% to 55%, during which consumption of the reducing agent remained acceptable.

## CONCLUSIONS

NOx storage technology is characterized by stages of adsorption of the nitrogen oxides in the catalytic converter and stages of desorption and of simultaneous deoxidization of nitrogen oxides to molecular nitrogen. By controlling these stages accurately, this technology has a far higher NOx reduction potential than continuous-operating DENOX principles.

It has been shown that NOx storage technology can also be used in the DI diesel engine with high NOx reduction potential. The NOx emissions of a 2.2 liter DI diesel engine were reduced by 80% at steady-state operating conditions by using a NOx storage catalytic converter. With the same configuration, a NOx reduction of 50% to 55% in the NEDC was verified repeatedly in the current development version of an experimental C-class vehicle.

The achieved NO<sub>x</sub> emission value are in the range of the boundary value discussed for the EURO IV norm of 0.25 g/km.

The influence of sulfur contained in fuel was shown as results from laboratory and engine testing. The formation and storage of sulfates in the catalytic converter, from the sulfur dioxide contained in the exhaust gas, leads to a decrease in the nitrogen oxide reducing capability of the catalytic converter. When using diesel fuel at 370 ppm sulfur content, this mechanism leads to a very fast decrease of the NO<sub>x</sub> reduction rate within a few hours of operation. Current technology dictates that sulfate regeneration in the diesel motor exhaust gas under normal driving conditions is not possible without excessive expenditure.

Even in the case of a sulfur content of 10 ppm in diesel fuel, over a longer period of time a decrease of NO<sub>x</sub> storage and reduction capability is probable. Therefore it is necessary to lower the sulphur content of Diesel fuel below 10 ppm to ensure the long term durability of the aftertreatment technology.

It can be concluded that the implementation of NO<sub>x</sub> storage catalytic converter technology depends very significantly on whether the problem of excessive sulfur content in fuel is solved and whether the operating life of the catalytic converters can be improved. In addition to meeting NO<sub>x</sub> limit requirements, it must also be guaranteed that current limits for particle, HC and CO emissions can be reliably fulfilled by optimizing the regeneration mode.

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